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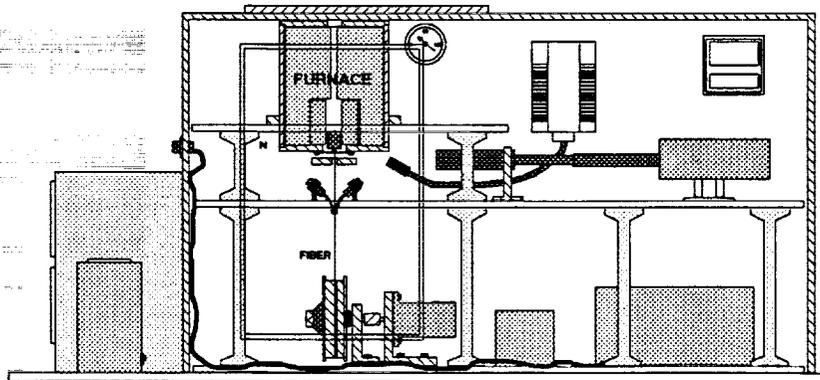
Fiber Pulling Apparatus Modification

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INTRODUCTION

A reduced gravity fiber pulling apparatus, henceforth FPA, has been constructed in order to study the effects of gravity on glass fiber formation. The apparatus was specifically designed and built for use on NASA's KC-135 aircraft. Four flights have been completed to data during which E-glass fiber was successfully produced in simulated zero, high and lunar gravity environments. In addition simulated lunar soil samples have been tested for their fiber producing properties using the FPA.

With NASA's commitment to a permanent manned presence in low earth orbit and eventual return to the moon, numerous studies have been performed in the areas of micro gravity and lunar materials processing¹⁻⁸. Continuous glass fiber processing of optical and single crystal fibers may be enhanced due to the absence of gravity forces⁹. In fact, a miniaturized fiber pulling apparatus for drawing single crystal core glass fibers in low earth orbit has been developed¹⁰. The Japanese also refined optical grade materials using a float zone furnace during the SpaceLab J mission in September of this year. On the lunar surface, abundant materials exist which can be used to produce structural materials¹¹. The use of lunar regolith for the production of structural materials could greatly reduce the cost for construction and long-term habitation of a lunar colony. One lunar product, fiberglass, promises ease of manufacture and wide applicability¹². Continuous fiberglass could be utilized as reinforcement in structural composites, including pressure vessels, glass cables and woven fiber insulation¹³.

Initially the FPA was developed under contract NAS8-36955, Delivery Order 113 over the period of March through September of 1991. At the end of that six month period it was determined that several improvements could be implemented into the apparatus to aid in formation and study of micron diameter glass fibers. Hence, a follow on contract was obtained to continue the research. The following provides the results of what has been accomplished.

DESCRIPTION OF THE FPA

Referring to figure 1, the FPA consists of a furnace and associated temperature controller, fiber take up reel and servo motor, video camera, fiber quenching and cooling system. These basic components are enclosed in a Plexiglas housing. Since this system flies aboard the KC-135 safety requirements dictated a containment system. There is a port for dry nitrogen gas so that the relative humidity of the apparatus can be reduced during fiber drawing. A hygrometer is located on the inside rear wall which reads relative humidity. The FPA is controlled by a data acquisition system. This system consists of an IBM AT industrial grade computer with Metrabyte model DAS8 eight channel 12 bit A/D board for recording data and controlling the servo winding motor and a video graphics overlay board. Also mounted within the rack is a super VHS video recorder, video monitor, keyboard, and associated control electronics. The video monitor displays the image

of the glass fiber as it is being drawn and overlays on it the graphical data. This data includes three axes accelerometer data from a set of Sundstrand model 700 accelerometers, furnace and bushing orifice temperatures, fiber winding speed in cm/sec, date and time.

The furnace canister in Figure 2 consists of an alumina tube wound with platinum 30% rhodium heating wire encased in alumina potting compound and subsequently in zirconia felt insulation. The furnace housing is made from 6061 aluminum. A pure platinum crucible or bushing with a single orifice is located within the platinum wound alumina tube. Bushings with a 2 and 4 millimeter diameter orifice are used. Three platinum/rhodium type S thermocouples are placed in the alumina tube to measure the furnace temperature and one at the bushing orifice to measure sample temperature. Temperature is controlled by a Eurotherm model 818S process controller located on the front wall of the FPA Plexiglas housing. An platinum vapor coated inconel sheet with a drilled center hole is located at the bushing orifice. This sheet holds the bushing in place and helps to reflect heat which would ordinarily be lost through the furnace bottom.

FPA OPERATION

With the furnace at the proper temperature glass sample in centimeter size pieces is loaded into the platinum bushing via a circular hole located at the top of the furnace assembly. The furnace operates on 120 VAC and draws up to 10 amperes of current. The maximum operating temperature of the furnace is 1600°C. The furnace is accessible through a door located at the top of the FPA. During heat up the FPA can be purged with dry nitrogen gas which can be run continuously during the winding operation. The winder is programmed for take-up speed in cm/sec as well as increments of increasing or decreasing speed. These increments are controlled by the UP/DOWN arrows located on the computer keyboard. The maximum winding speed is 1000 cm/sec and the increments can be adjusted from tenths of a cm/sec to 100 cm/sec. Once the sample has reached the appropriate drawing temperature and thus viscosity, a ceramic rod or large quartz fiber is used to manually draw the fiber from the bushing orifice to the take-up reel. The reel is a six inch diameter aluminum wheel with double sided tape applied to the take-up surface. Once drawing has begun, Fiber diameter can be controlled by varying the winding speed and sample temperature. A simple fiber coating apparatus can then be brought in during the winding operation to coat the fiber and thus protect it from abrasion and humidity. It consists of two hard felt pads which are connected to a supply of polyvinyl alcohol. Capillary action draws the PVA down into the felt pads through which the glass fiber runs. A video camera is positioned such that, during the fiber pulling operation, a continuous record of the fiber jet as it exits the bushing orifice can be made. The TVGA graphics overlay board located within the computer adds the parameter data to the video image prior to being recorded on the S-VHS VCR. Also a microphone allows voice description of the experiment conditions to be recorded.

PLEXIGLAS HOUSING

ACCESS DOOR

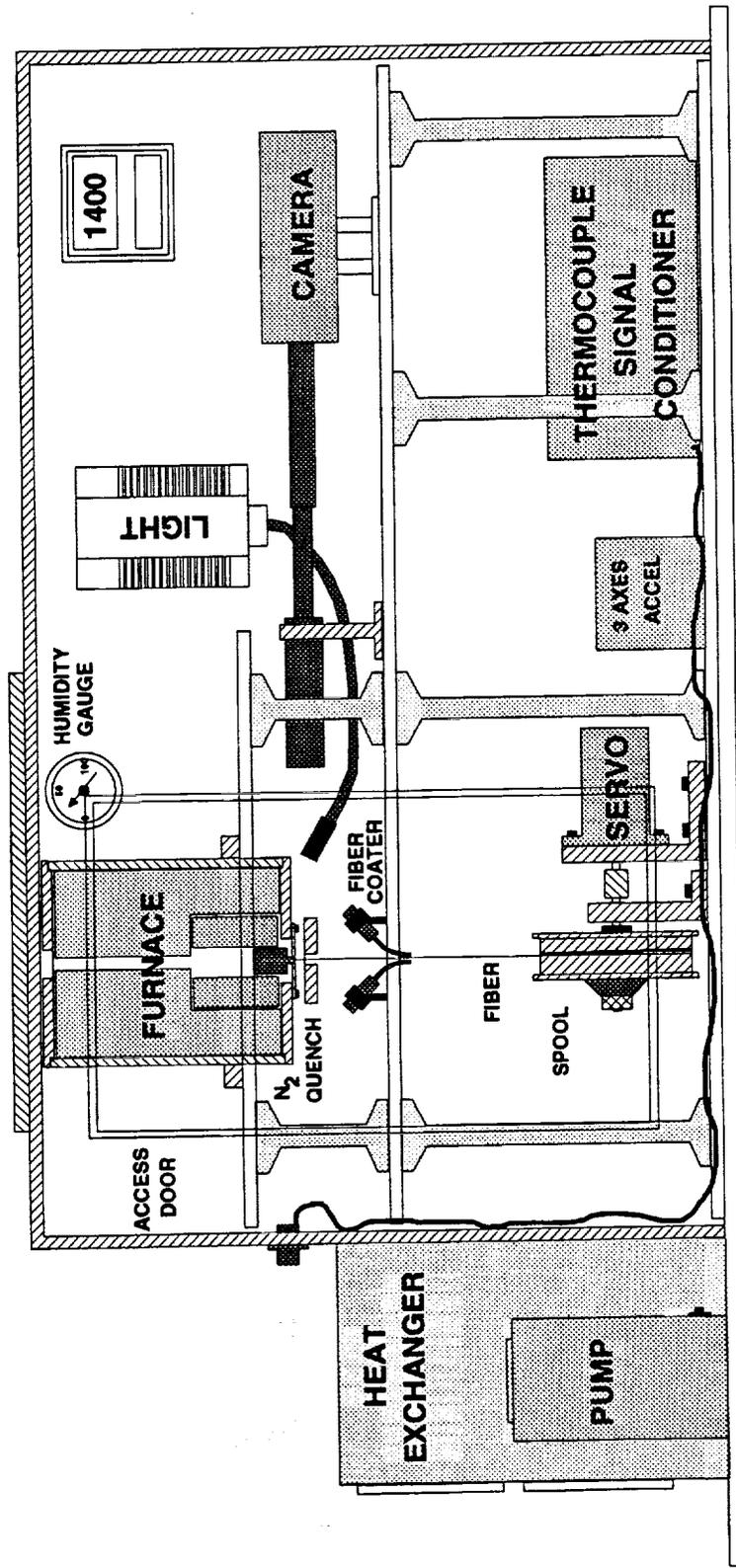


FIGURE 1: Diagram of the Glass Fiber Pulling Apparatus

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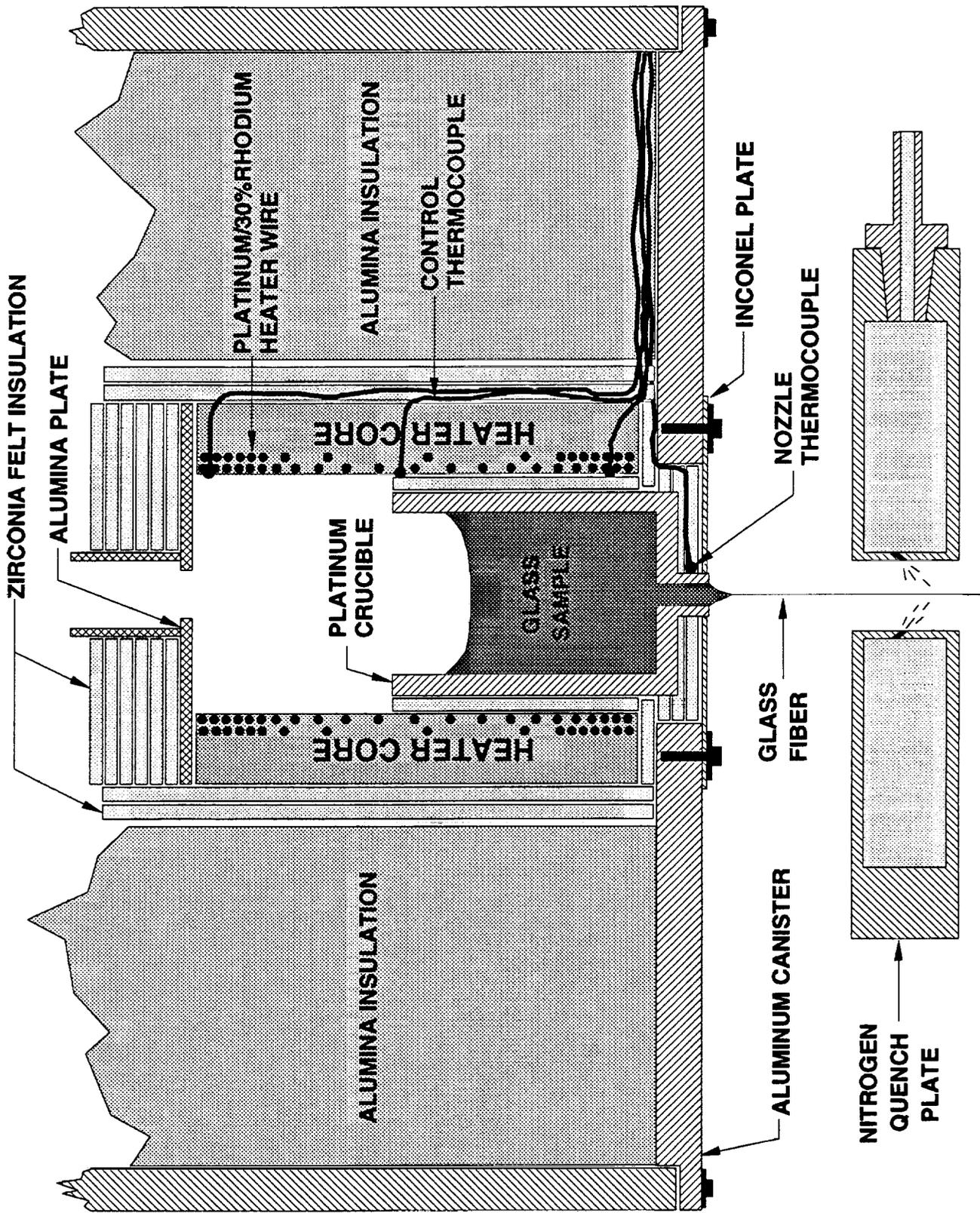


FIGURE 2: Cross section of furnace core and nitrogen quench block.

There are two options available to quench the fiber after it exits the furnace. One option is a brass annulus with a 1 mm diameter hole drilled in the center to allow the fiber to pass through. This annulus acts as a quench plate. A water/antifreeze mixture is circulated through the plate via a heat exchanger pump. This plate would be used if one were pulling a fiber above 1200°C. A quench is required so that the fiber is cool enough to allow winding. This option is also useful if a fiber is being produced from a material which has a tendency towards recrystallization such as in the case of the lunar simulant. Quenching the fiber rapidly helps prevent nucleation events which can adversely affect fiber properties. The other option involves using dry nitrogen gas to quench the fiber. In this method, a brass annulus with a 13 mm diameter hole in the center and twenty 0.7 mm holes arranged around the inside perimeter is used to direct nitrogen gas onto the fiber as it is pulled. This not only quenches the fiber but aids in the drawing process.

During a reduced gravity maneuver in the KC-135 aircraft, the acceleration due to gravity can be as low as 0.001g and as high as 2g. Thus, the FPA was constructed to withstand the loads seen during the parabolic maneuvers, as well as 9g emergency crash landing loads. This reason, plus on-board safety considerations, is what dictated overall designs of the FPA system.

RESULTS

Two types of glass material were used in this investigation: E-glass and lunar simulants. E-glass is a silica based glass with well characterized fiber forming characteristics. It was used as the control sample. Two types of lunar simulant were also tested for their fiber forming characteristics. The lunar simulant material was obtained from Dr. Paul W. Weiblen of the University of Minnesota Space Science Center. Using a plasma processing system, raw soil collected from Minnesota is modified in terms of weight percent composition to more closely match the lunar material collected during the Apollo era. The two lunar simulants are identified as MLS-1 and MLS-2. MLS-1 is representative of the mare basalt and MLS-2 is of the lunar highlands. The following table provides a weight percent break down for comparison. It should be pointed out that the average percent element concentration for the MLS-2 if prior to the plasma processing technique. Once processed the elemental percents more closely match that of the lunar highlands regolith. Post plasma processing element analysis was in progress and not complete at the time of this report.

Table 1: Simulant vs. genuine lunar regolith vs. E glass comparison

COMPOUND	MLS-1 AVERAGE %	MARE ¹ BASALT	MLS-2 ² AVERAGE %	LUNAR ³ HIGHLANDS	E-GLASS AVERAGE %
SiO ₂	43.86	42.55	50.63	43.90	55.00
TiO ₂	6.32	7.71	0.07	0.50	-
Al ₂ O ₃	13.68	13.47	30.66	28.80	15.0
FeO	13.40	15.16	0.94	4.90	-
Fe ₂ O ₃	2.60	-	0.28	0.00	0.50
MgO	6.68	7.98	0.08	0.10	0.50
MnO	0.198	0.208	0.59	5.70	-
CaO	10.13	11.99	13.32	15.60	22.00
Na ₂ O	2.12	0.445	2.88	0.50	-
K ₂ O	0.281	0.147	0.25	0.10	-
P ₂ O ₅	0.20	0.140	0.16	-	-
H ₂ O	-	0.00	0.98	0.00	-
B ₂ O ₃	-	-	-	-	7.00
CO ₂	0.0015	-	-	-	-

¹ Apollo sample number 10084.

² Prior to plasma processing.

³ From Taylor, S.R., 1975, Lunar Science: A Post-Apollo View, Table 5.4, p. 216, Pergamon.

A. RESULTS USING E-GLASS MATERIAL

During the initial stages of testing and subsequently throughout the project E-glass was used to establish base line parameters in the operation of the FPA system. Since E-glass easily forms glass fibers it was chosen to be the reference material. The E-glass was supplied in 25 mm marble form. These marbles were remelted at 1400°C for 24 hours in a platinum crucible and poured onto an aluminum block into droplets approximately 10 mm in diameter. The droplets were placed into the FPA furnace and heated to various temperatures and fibers drawn at various speeds to determine the operating characteristics of the system.

In December of 1991 E-glass was processed aboard the KC-135 during various gravity levels. Of particular interest during this experiment was the shape of the jet zone and the effect that gravity played upon that shape. Referring to figures 4,5, and 6 the influence of gravity can be clearly observed. The furnace was heated to 1175°C which gave a orifice temperature of approximately 1100°C. Fiber was continuously pulled throughout each maneuver. During the 1.8 g portion of the maneuver the fluid jet was observed to be 4mm in diameter at a constant winding speed of 30 cm/sec. As the g load was decreased the fluid jet diameter was seen to decrease steadily to a minimum value of 3 mm at 1/6 g (lunar gravity). This affected final fiber diameter as a function of g-load. At 1/6 g the final fiber diameter was measured to be 100 micrometers, at 1 g, 160 micrometers and 240 micrometers at 1.8 g.

The three images of the jet zone shape exiting the crucible orifice were captured off the S-VHS tape used to record the data during the flight. The legibility of the text written by the TVGA adapter with the genlock daughter board and subsequently recorded on the S-VHS tape was

difficult to read. The text was therefore enhanced by using TRECWare¹⁴ Digital Image Processing software running on an Imaging Technology Inc. PC vision Plus frame grabber. The selected frame was split into even and odd fields of 240 lines each. The fields were vertically magnified to a full frame size of 480 lines. The high frequency nature of the digital magnification preserved all the characteristics of the original image frame while at the same time enhancing the legibility of the overlaid text.

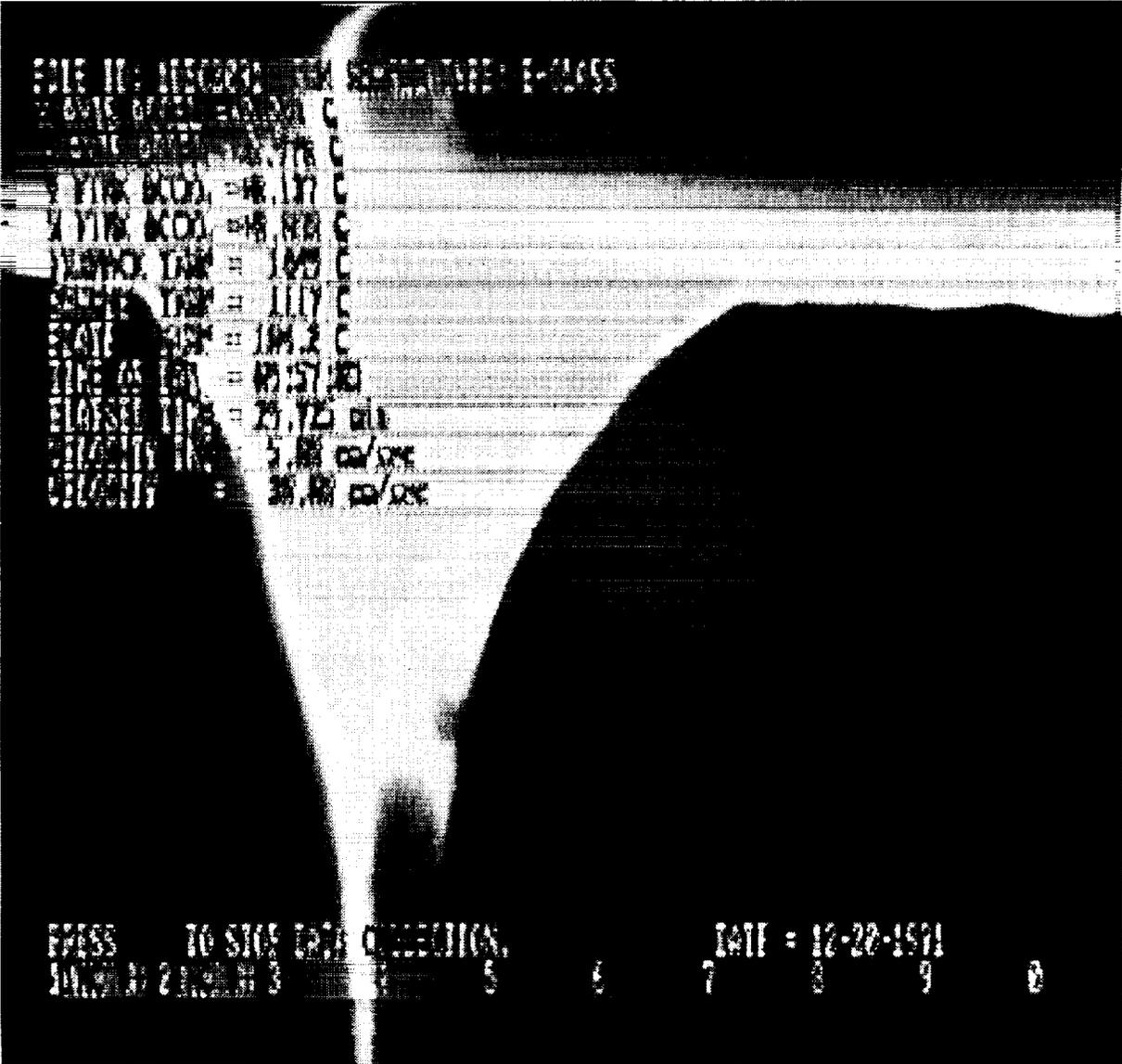


Figure 3: Shape of jet zone of E-glass during normal 1 g level

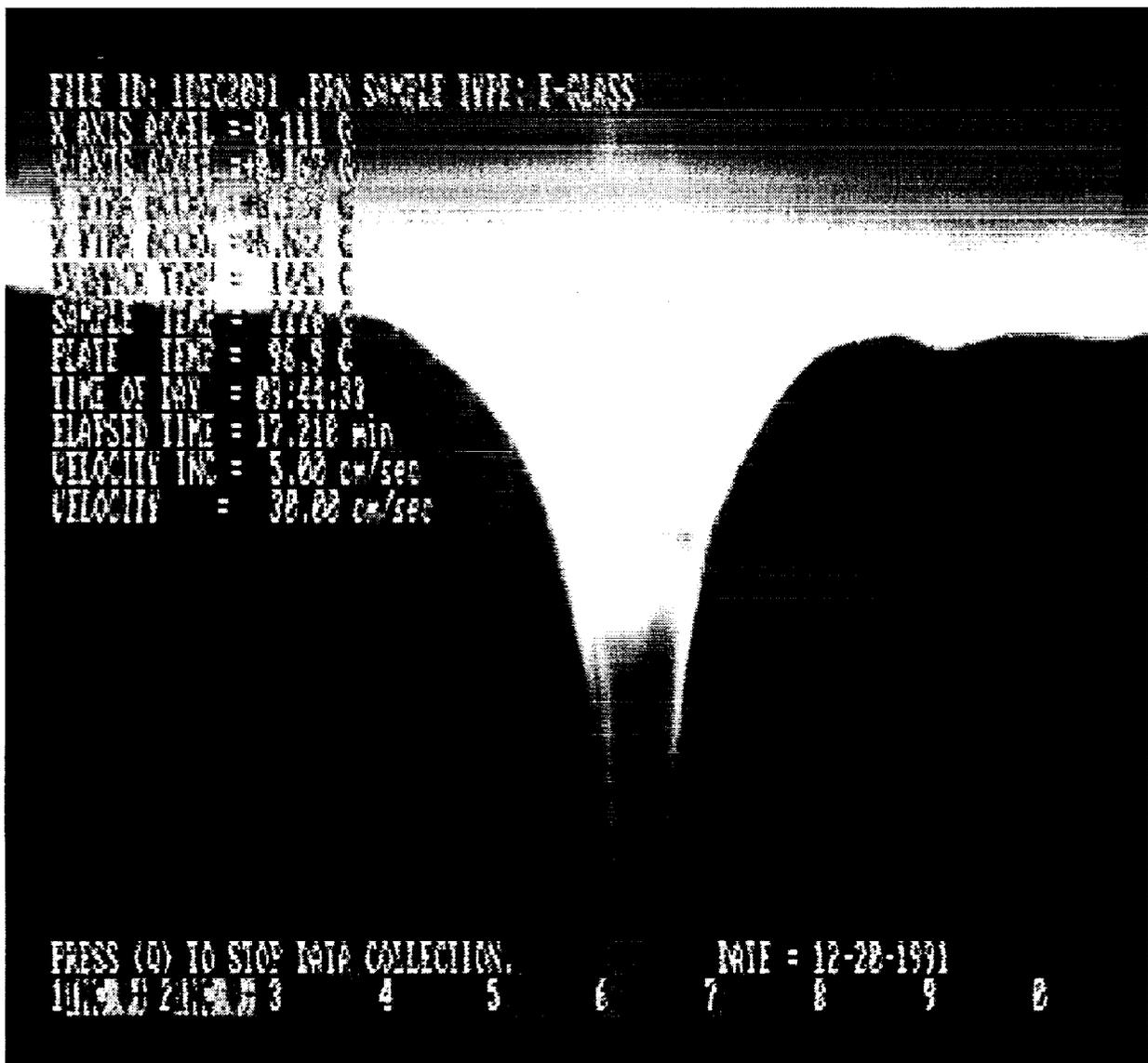


Figure 5: Shape of jet zone of E-glass during 1/6th g (lunar) level

During the flight it was determined that if the winding speed was increased above 30 cm/sec the fiber would break approximately one inch below the orifice due to attenuation of the fiber jet at 1/6 g. The strength of the fiber produced during the flight was not tested since a coating mechanism had not yet been developed. Without a coating to protect the fiber its strength decreases rapidly due to exposure to moisture and abrasion.

B. RESULTS OF LUNAR SIMULANT MATERIAL

The Minnesota lunar simulant was received in a granular form. It was then remelted to 1400° C in platinum crucibles using a box furnace and allowed to soak for 24 hours. The homogenized material was then poured out into large droplets for use in the various furnaces.

In processing the lunar simulant it was soon determined that it had poor fiber producing properties. Numerous ground runs using four different types of furnaces failed to produce good results in terms of pulling a continuous fiber longer than a few feet. Initial tests using MLS-1 indicated that the working range for being able to pull a fiber was within only a few degrees of 1240°C. In addition the fiber once drawn had to be quenched quickly to prevent recrystallization and attenuation. Dopants were added to perhaps improve the fiber pulling properties. In several batches SiO₂ was added to MLS-1 to bring the percent up to 50 wt%, 55 wt%, and 60 wt%. Differential thermal analysis (DTA) was performed which indicated that the cooling curve was smoothing out with the higher percent content however the recrystallization problem increased.

In another batch B₂O₃ was added to provide a 10 wt% and 20 wt% doped MLS-1 material. This resulted in much better fiber pulling properties. DTA's were also performed to establish the melting and recrystallization points of the various batches. One additional MLS-2 batch with 8 wt% B₂O₃ was determined to be the optimum mixture. Several successful runs were performed using both the ground based furnace and the flight unit. The finest run occurred with a furnace control temperature of 1350°C and a orifice temperature of 1320°C. This resulted in a continuous fiber of high quality with no recrystallites. Fiber diameter reached a minimum on 7.5 micrometers with a drawing speed of 755 cm/sec. During the drawing process nitrogen gas was used to quench the fiber and a coating of PVA was immediately applied. The PVA coating was a solution of 5% PVA in deionized water. Tensile testing of the fiber was still pending at the time of this report.

CONCLUSIONS

Based on the results of the research effort the two types of lunar simulants tested do not lend themselves very well to pulling a continuous fiber when used in their natural state. This is due to the narrow working range of the material and its tendency to easily recrystallize back into a "rock" form. Doping the lunar material with 8 wt% B₂O₃ greatly improves its ability to produce a continuous fiber. In its natural state however lunar material could still be used to produce short

fibers using a spin melt method similar to a cotton candy machine. This could easily provide insulation bats and short random fibers for composite reinforcement

FUTURE WORK

With the present FPA flight system being fully operational, research is continuing in the field of glass fiber pulling. Additional studies are needed in the field of fluid dynamics and the role that gravity plays upon the formation of fibers. It is also anticipated that optical grade glasses will be processed to determine if gravity has an effect upon the quality of the fiber.

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